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REPORT

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NORMAL LEAD STYPHNATE SUITABLE FOR AUSTRALIAN
PRODUCTION OF LOW SENSITIVITY CC PRIMERS

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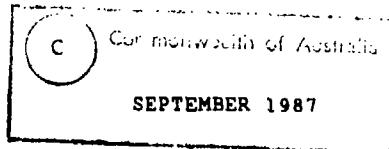
R.J. Spear, L.D. Redman and V. Nanut

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NORMAL LEAD STYPHNATE SUITABLE FOR AUSTRALIAN
PRODUCTION OF LOW SENSITIVITY CC PRIMERS

R.J. Spear, L.D. Redman and V. Nanut

ABSTRACT

Normal lead styphnate types RD1302, RD1303M and RD1367 were assessed for their suitability for production and use in Australian manufactured low sensitivity CC primers. Recommendation was made that type RD1302 should be used. The sensitivity penalty incurred by using small particle size lead styphnate has been demonstrated. Pilot lot production of primers at MFF St Marys using selected production RD1302 has verified suitability for the intended application. Australia ↙

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NORMAL LEAD STYPHNATE SUITABLE FOR AUSTRALIAN
PRODUCTION OF LOW SENSITIVITY CC PRIMERS

1. INTRODUCTION

The 6-barrel Vulcan M61A1 rapid fire gun forms part of RAAF's F/A-18 weapons system and is the firing system for the RAN's Phalanx anti-missile close in weapon. Although the ammunition used for these two applications is different (Raufoss 20 mm M70A1 Multipurpose Concept and TP Ammunition (F/A-18) and 20 mm (DS) Mk 68 Mod 1 (Phalanx)) both are primed with a conducting composition (CC) primer of US design, the M52A3B1. The M52A3B1 is a very sensitive primer [1,2] which is particularly susceptible to initiation when subjected to high intensity RF signals (RADHAZ) which are typically generated by Radar and Telecommunications facilities at airfields and onboard ships. Operational restrictions currently apply to Phalanx because of this susceptibility. A programme to develop a CC primer which met the performance specifications of the M52A3B1 [3] but was of substantially lower sensitivity to RADHAZ (higher threshold firing energies) has been proceeding at MRL for about 2 years [2,4].

One potential problem noted during development of the low sensitivity primer has been the availability of suitable normal lead styphnate to be used in admixture with Tintacarb 140, the conducting increment. All the developmental work was carried out on a single batch prepared at MRL, nominally type RD1303M, average particle size 115 µm. MFF St Marys can currently produce two types of normal lead styphnate, RD1302 and RD1303M. The RD1303M as produced is usually very small average particle size (30-40 µm). Our studies [4] and an earlier study [5] had both indicated that decrease of lead styphnate particle size led to increased primer sensitivity.

We therefore undertook to identify a procedure for consistently preparing normal lead styphnate of suitable particle size, and to make recommendations to MFF St Marys on what type would be most suitable for production of the primers. This study, and subsequent pilot lot production at MFF St Marys, forms the basis of this report.

2. NORMAL LEAD STYPHNATE TYPES

Five types of normal lead styphnate have been described in detail to pilot plant or full scale production. They are listed below with broad descriptions of their development history and properties.

- RD1302 [6,7]: Developed for general use in detonators. The principal requirements were that the material be free-flowing, high bulk density, relatively uniform particle size and dust-free. Typical particle sizes were around 100 μm but could be tailored over a wider range.
- RD1303 (O and M) [6,7]: Developed for percussion and electric cap compositions, and fuzehead compositions. This material was of considerably smaller particle size than RD1302 to provide the required sensitivity coupled with good mixing properties. The RD1303M modification gave a material of reduced electrostatic sensitivity.
- RD1316 [7,8]: Lead styphnate co-precipitated with 1% methylcellulose to give a granular free-flowing product. The main use was in fast burning delays in fuzes and detonators. Typical particle sizes were around 200 μm .
- RD1318 [7,9]: The β -polymorphic form, suggested to have reduced susceptibility to electrostatic initiation.
- RD1367 [10,11]: A cost-effective product developed by modification of the basic RD1302/3 process. Yields are higher, concentrations higher hence volumes lower, and impure styphnic acid can be used as feedstock.

Neither RD1316 nor RD1318 have been adopted for UK service use [12] and RD1316 does not appear to be of a desirable form for CC primers. RD1367 has been adopted for UK service use [12]. The decision was therefore made to proceed with investigations on RD1302, RD1303M and RD1367.

3. RD1302/3 AND RD1367 CHEMISTRY

All three production processes involve initial reaction of aqueous styphnic acid and magnesium carbonate to form an aqueous solution of magnesium styphnate. This solution is then subsequently reacted to form the resultant normal lead styphnates.

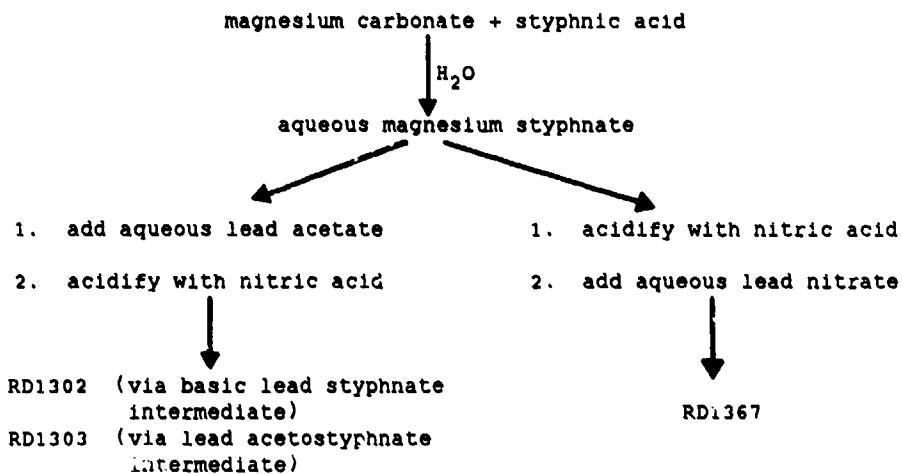
In the case of RD1302 and RD1303 the magnesium styphnate solution is reacted with aqueous lead acetate followed by acidification with nitric acid

to give the final products. The differences between RD1302 and RD1303 arise from differences in concentrations, addition rates and times, and final pH [6]. The difference is in fact more fundamental than may be apparent: although Taylor [6] stated that both processes involved the intermediacy of "basic lead styphnate" which was subsequently converted upon acidification to the normal salt, this is only true in the case of RD1302 [13]. The conditions for RD1303 differ sufficiently in pH and other concentrations that a second intermediate, lead acetostyphnate, is formed which subsequently converts to the normal salt upon acidification [13]. The differences between RD1303 O and M arise solely from differences in addition time of the nitric acid in the final acidification step.

The RD1367 process employs a different reaction scheme and more concentrated reagents. The aqueous magnesium styphnate solution is first acidified with nitric acid to give a known concentration of free styphnic acid (FSA), then reaction with aqueous lead nitrate gives the final product [10].

The reaction schemes are shown below as Scheme 1.

Scheme 1 : Reaction Sequences for RD1302, RD1303 and RD1367



4. RESULTS AND DISCUSSION

4.1 RD1367

The decision was made to study RD1367 first because previous experience at MRL [14] suggested that the process could be tailored by control of FSA concentration to yield a product of the desired particle size.

Batches were prepared with FSA concentrations, produced by acidification and determined titrimetrically, from 0 (basic) to 20 g/L. The results for FSA, particle size and yield of the products are detailed in Table 1.

The British Patent on RD1367 [10] includes descriptions of preparations using FSA from 0-15 g/L. Recommendation is made therein that the process is preferentially carried out at 1-5 g/L FSA, resulting in typical yields of 93% of theoretical. At higher FSA values the yield reduces substantially, eg 73% at 14.8 g/L FSA [10]. Jenkins [11] has given some data for the product from the optimised RD1367 process; bulk density 1.5 ± 0.15 Mg/m³, average particle size 65 µm with 80% between 20-130 µm. The FSA at which this process was carried out was not stated.

Previous studies at MRL [14] showed that there was a relationship between RD1367 particle size and FSA for preparation from crude styphnic acid. Inspection of photomicrographs showed that increased FSA gave decreased particle size.

Examination of the results in Table 1 shows that most mean particle sizes (see explanation Section 7.3) fall in the range 60-90 µm. There is a general trend, with the single exception of the FSA 10.0 g/L result, to increased particle size with increasing FSA. This is the opposite to that observed previously [14]; there is no obvious answer for this discrepancy. Yields are generally lower than quoted in ref [10] but no attempt was made to optimise this parameter. Although there is not the steady decrease in yield that was observed for FSA > 5 g/L [10], yield had decreased significantly at 20 g/L FSA (Table 1). Yield at the lowest FSA level (basic, Table 1) was substantially lower due to losses in decanting from the messy final mix; there have always been problems with preparation of RD1367 at very low FSA concentrations [11,13].

Although the results observed differ somewhat from earlier studies [10,11] it should be stressed that the styphnic acid used here was considerably different; the RD1367 process was developed to use a particular crude styphnic acid. Any process being trialled for eventual production at MF St Marys must use their styphnic acid which can be supplied either crude or recrystallised. All normal lead styphnate production processes display a marked dependence on the type of styphnic acid used as feedstock [13].

4.2 RD1303M

The batch of normal lead styphnate (14/78) used in the primer developmental work was nominally of type RD1303M. A series of batches using the standard method [7] was prepared and results are listed in Table 2 for both yield and particle size. Particle size results for 14/78 and a production batch of "RD1303M" from MFF St Marys are listed for comparison.

None of the experimental batches was as large in particle size as 14/78, but mean particle sizes covered a relatively narrow range and showed reasonable reproducibility. Note the substantially smaller particle size of the MFF production batch; the process at MFF had presumably been optimised for mixing, consistent with its major use as a component in multicomponent mixes.

4.3 RD1302

Preparation of RD1302 was carried out using both the general method described in [6] and modified procedures. The complete results for all batches are listed in Table 3. Particle size measurement on production batches from MFF St Marys are included for comparison.

The initial batches (27/86 and 28/86) were prepared at a stirrer speed of 360 rpm and had mean particle size 80-90 μm . Stirrer speed has a quite pronounced effect on particle size [6]; reduction in stirrer speed tended to give larger crystals but, if the speed was reduced too much, settling of the intermediate occurred and the production process suffered. We accordingly prepared three batches (31/86, 33/86, 34/86) at stirrer speed 180 rpm (Table 3).* All these batches had mean particle size equal or greater than those prepared at the higher stirrer speed. Particle sizes showed some variance between the three batches with the highest at about 123 μm . Reduction of the stirrer speed to 100 rpm (batch 32/86) gave no decrease in processability but particle size was a little smaller.

A further modification which was suggested [13] was the addition of acetic acid, which had been observed to give a larger, less aggregated product with smaller particle size spread. Carrying out the RD1302 process using lead acetate acidified with acetic acid at 1.0 mL/L concentration (batch 29/86) afforded no change relative to batches 27/86 and 28/86 while at concentration 1.8 mL/L the product exhibited a bimodal particle size distribution (batch 30/86, Table 3). The maxima occurred at 74 and 137 μm and represented approximately equal masses under each envelope. Further use of acetic acid was discontinued on the basis of this result.

* All stirrer speeds quoted refer to the MRL 600 mL pan with the exception of the 35 rpm cited in Section 5 and the procedure in Appendix A which refer to MFF St Marys production equipment.

4.4 Variation of Primer Sensitivity with Normal Lead Styphnate Type

A necessary precondition for selection of the most suitable type of normal lead styphnate for production was that sensitivity of the resulting primers was not increased significantly over those filled with RD1303M Batch 14/78 as used in the development studies. Batches 16/86 (RD1367) and 34/86 (RD1302) were chosen as representative of each of these types prepared during the course of this study, and both possessed mean particle sizes which were relatively large. Admixtures with Tintacarb 140 (96:4, 75 mg) were prepared and filled into M52A3B1 empties followed by DEFA priming mix (100 mg). Full experimental details are given in Section 7. A third series of primers was prepared from a production lot of RD1303M ex MFF St Marys chosen because of its small particle size (approximately 30 µm). The object here was to obtain a quantitative measure of the penalty incurred in increased sensitivity due to small mean particle size lead styphnate.

The primers were measured for resistance at pressing and after 2 weeks, then energy sensitivity was determined using Bruceton analysis [15] as an initial screening (25 primers) followed by Probit analysis [16] (125 primers). Results are detailed in Table 4 and are compared with results from primers prepared from RD1303M batch 14/78.

Both sets of primers prepared from RD1302 and RD1367 exhibited very similar resistance and energy sensitivity. Energies for 50% firing probability were increased over the corresponding Batch 14/78 primers, but were not strictly comparable (see footnote d, Table 4). The penalty for using small particle size lead styphnate is readily seen from the results for RD1303M Batch 34 where the 50% firing energy was about half that for the RD1302 and RD1367 batches but the threshold firing energy (0.1%, 95% conf.) was well below those for the other two primers. It should be noted that both 50% and threshold firing energies for RD1302 and RD1367 cited in Table 4 were approximately double those observed for RD1303M Batch 14/78 in DEFA primer bodies [2]. Resistances were also considerably lower [2] because of the different design of the M52A3B1 and M52 DEFA primer bodies. It is unfortunate that all the development work could not have been carried out in M52A3B1 bodies but these were not available at that time.

The conclusion from these results is that any of the normal lead styphnates RD1302, RD1303M or RD1367 would be suitable for use in the primers provided particle size is sufficiently large. A small particle size material could not be substituted without paying a considerable penalty in increased sensitivity.

5. RECOMMENDATIONS ON WHICH TYPE OF NORMAL LEAD STYPHNATE SHOULD BE PRODUCED FOR PRIMER MANUFACTURE

Our results for types RD1302, RD1303M and RD1367 show that each can potentially produce a suitable product. A picture of particle size distribution can be seen from the results below:

RD1302 34/86 Median 80 µm, 16% < 58 µm, 16% > 112 µm
RD1303M 3/86 Median 90 µm, 16% < 57 µm, 16% > 130 µm
RD1367 16/86 Median 70 µm, 16% < 34 µm, 16% > 123 µm

Clearly the batches are quite similar and could all equally be selected on the basis of particle size. The decision was made to recommend to MFF St Marys that type RD1302 be used in primer production. The reasons for this were as follows:

- (i) The RD1302 process has been designed to produce a larger, ie dust free, product [6]. Our results indicated that a reproducible material of the required particle size was readily achievable.
- (ii) MFF St Marys have extensive experience with the RD1302 process.
- (iii) The RD1303M process was designed to yield a small particle size product, and our experience with MFF production material suggested that a small particle size product was normally obtained. On our small batch size we normally obtained larger particle sizes (Table 2).
- (iv) The RD1367 process was designed to use as feedstock a type of styphnic acid not produced at MFF. In addition our results indicated that some variation in particle size between batches would be expected, and considerably more development effort would be required to specify an optimum FSA level.

An outline of the MFF production process for RD1302 is given as Appendix A. Since our experience with RD1302 as manufactured by MFF was that the product tended to small particle size (see Table 3, but see later comments) we suggested the following changes to the production process which we believed would facilitate production of a larger particle size RD1302 product.

- (i) Change stirrer speed to 35 rpm.
- (ii) Stir for 5 minutes after addition of lead acetate solution.
- (iii) Do not tilt pan.
- (iv) Do not cool to 36°C but complete the final 15 min stirring after addition of nitric acid without heating.

MFF prepared a batch with these changes. However the scaleup from MRL to MFF production facilities of 1:123 (compare section 7.2.1 to Appendix A) was too large and the resultant batch was a failure; the principal problem was sedimentation at the reduced stirring rate. MFF therefore changed their RD1302 process such that particle size of the product was increased. The batch was successful and is referred to as batch 4 in the following text.

Two pilot production runs of approximately 250 primers each, designated Mix A and Mix B, were filled at MFF. Mix B used RD1302 production batch 4 (see above) in the conducting increment. Mix A used a production batch (batch 2753) especially selected as being of appropriately large particle size and high bulk density. Mr B. Alsop [17] has stated that it was common practice during production of M52 DEFA primers to select larger particle size batches of RD1302 from general production to use in the conducting increment; the MFF lead styphnate process gives product of reproducible bulk density but particle size can show considerable batch to batch variation. The primers were filled with a 96:4 admixture of lead styphnate with Tintacarb 140 (75 mg) as conducting increment and DEFA priming mix (100 mg) as priming increment. Production staff indicated their approval with the ease and reproducibility of the process.

120 primers each from mix A and B, together with samples of the lead styphnate used in the conducting increments, were forwarded to MRL for assessment. A second batch of RD1302 (batch 2753) selected as potentially suitable was also forwarded for comparison. Results for particle size measurement on the lead styphnates and sensitivity and functioning time measurements on the primers are detailed in Table 5.

Both lead styphnate batches used in the production primers were of desirable mean particle size and distribution, as was batch 2753. Resistance of the primers was a little higher than those produced at MRL (Table 4) but of narrow standard deviation (less than 10% of mean). Surprisingly, energies for 50% functioning probability were nearly double those we obtained (Table 4) which resulted in about 15% failure to fire in the functioning time test [3,4]. Functioning times of primers that fired were well within specification and were very reproducible.

The energy sensitivity of the production primers was unexpected and probably results from one or more of the following:

- (i) More efficient mixing of the conducting mix due to use of a jelly mould mixer rather than our method of cold-mixing.
- (ii) Variation between our batch of Tintacarb 140 and that used by MFF.
- (iii) Differing pressing conditions either in the filling increments or on subsequent capping of the primers. This was probably the main reason for the slightly higher resistances.

The key observation is that insensitive primers can be successfully and reproducibly filled at production. Slight reduction of Tintacarb 140 content will bring the energies down to acceptable levels and this has subsequently been achieved in a larger pilot lot. Details will be given in a subsequent report.

6. SUMMARY/CONCLUSIONS

Investigation of normal lead styphnates for suitability for production and use in low sensitivity conducting composition primers has resulted in a recommendation that type RD1302 be used in the conducting increment. Production capability to produce material of the required particle size range is acceptable although batches may still need to be selected on the basis of particle size, as was the case for M52 DEFA production. The use of small particle size lead styphnate is unacceptable because it leads to a very wide range of sensitivity which results in low threshold firing energies.

Production of primers has been proved. Some adjustment in formulation will be necessary to decrease firing energies such that the all-fire energies are decreased and there are no failures in the functioning time specification test. Substitution of smaller particle size lead styphnate would result in lowering of 50% and all-fire energies. However the wide range of energies which would result would lead to unacceptably low threshold firing energies. A small decrease in Tintacart content has already partly overcome this problem.

7. EXPERIMENTAL

7.1 Materials

Lead acetate, lead nitrate, magnesium carbonate, nitric acid and acetic acid were all laboratory grade commercial materials which were used without further purification. Styphnic acid was from a production batch (Batch 185) ex MFF St Marys, supplied recrystallised and under water. The material as received was initially dried under suction then air-dried at ambient temperature and used without further purification. Distilled water was used for all preparations.

7.2 Preparation of Normal Lead Styphnate

All preparations carried out at MRL were conducted on a 600 mL remote facility designed and manufactured at ERDE Waltham Abbey (now RARDE Waltham Abbey). This facility has been described in detail in earlier publications both from MRL and ERDE.

7.2.1 Type RD1302

The method used was exactly as described in ref [7] except that the scale was reduced by a factor of 20, ie styphnic acid (25.6 g). Stirrer speed was adjusted to the desired rate after addition of the magnesium styphnate solution and monitored during the course of addition of the lead acetate and nitric acid solutions. The modifications using acetic acid (1.0 or 1.8 mL)

were carried out by addition to the magnesium styphnate solution. The product was washed with water by decantation, then ethanol, and finally air-dried.

7.2.2 Type RD1302M

The method used was exactly as described in ref [7] except that the scale was reduced by a factor of 22.5, ie styphnic acid (24.6 g). Stirrer speed was 360 rpm and the product was isolated in the manner described above for RD1302.

7.2.3 Type RD1367

A solution of magnesium styphnate was made up from styphnic acid (242 g) and magnesium carbonate (90 g) in water (950 mL) by stirring at 75°C. Free styphnic acid (FSA) concentration was determined on the above solution by titration using standardized (potassium hydrogen phthalate) 0.1 M sodium hydroxide solution with bromothymol blue indicator.

A portion of the standardized magnesium styphnate solution (120 mL) was placed in the reaction vessel and then concentrated nitric acid sufficient to give the desired FSA level was added. The reaction was heated to 80°C with stirring (360 rpm) then an aqueous solution of lead nitrate (120 mL of a 331 g/L solution) was run in over 15–20 min. The reaction was stirred for a further 10 min, cooled to 50°C and the product isolated as described above for RD1302.

7.3 Particle Size Measurements

Particle sizes were determined using a Malvern Particle Size Analyzer Model 2600/3600. The samples as slurries in HPLC grade chloroform were dispersed using energy from an ultrasonic bath for 1 minute, then transferred to an optical cell and placed in the laser beam path.

Initially samples were prepared by various means including the slurry method and the coning and quartering method which are both described by Allen [18]. However the preferred technique of sample preparation was to divide about 1 g of the powder into small fractions (10 to 20 mg) by using a rotary sample divider [18] constructed at MRL. Sample splitting of powder slurried in chloroform was preferred to splitting the dry powder due to the hazard of electrostatic initiation.

The principle of the Malvern is based upon Fraunhofer diffraction of a laser beam by small particles. The data thus generated is automatically collected and subsequent data reduction is performed using computer programmes supplied with the instrument. The results are presented as total mass per sampling band (of particle size range), not total number of particles per sampling band. The particle size distributions are obtained by fitting the light energy distribution to several particle size distribution models. The two models which were found to be most applicable were

- (i) A "model-independent" 16 parameter fit over 15 size bands
- (ii) A two parameter Rosin-Ramler distribution model

Both models gave close agreement for most of the samples over most of the distribution range, differing significantly only at the extremities of the particle size distribution. Where the particle size distribution was bi- or multi-modal only the "model-independent" method was suitable.

7.4 Explosive Fillings

Normal lead styphnate and Tintacarb 140 (through 106 μm sieve) were mixed in the weight ratio 96:4 by fold mixing. DEFA priming mix was prepared as described previously [4]. Production mixes differed in that they were blended in a "jelly mould" mixer.

7.5 Filling of Primers

The M52A3B1 primer bodies were filled with two increments, a conducting mix and a priming mix, as described in ref [4]. The primers were then capped with a paper disc and washer.

The first increment consisted of 75 ± 2 mg of conducting mix and was pressed at 123.5 MPa (400 kg dead load). The second increment of DEFA priming mix (100 ± 5 mg) was pressed at the same load. Both increments were pressed with a stepped drift which was used to reduce the weight of material needed for the first increment. An 8 mm paper disc was then pressed into place by an interference fit washer using a hand press.

Production primers from MFF St Marys were prepared in a similar manner.

7.6 Performance Testing

Resistances were measured using an Hioki 3208 Calcu Hi Tester.

Energy sensitivity was determined using the capacitor discharge firing box described previously [4]. Small batches (20-25 primers) were assessed using the Bruceton staircase method [15] using $0.1 \mu\text{F}$ /variable V in constant LogE increments. Larger samples (100-125 primers) were assessed by initially performing a Bruceton type test then extending to the Probit method [16]. Data reduction was by computation.

Production primers (Mix A and B) were (inadvertently) tested using a $0.2 \mu\text{F}$ capacitor.

8. ACKNOWLEDGEMENTS

The technical assistance of Mr R. Porteous for preparation, filling and testing was invaluable. Mr J.R. Bentley gave freely of his time and his many suggestions were of great help during the course of this study. Staff at MFF St Marys were also of great assistance and we would like to single out Mr G. Siddiqui for modifications to the MFF RD1302 process, Mr J. Denton for preparation of lead styphnate, and Mr B. Alsop for the contributions they made.

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TABLE 1

**Results for Preparation of Normal Lead Styphnate
Type RD1367 Performed at Various Free
Styphnic Acid (FSA) Concentrations**

Data for RD1367 Product		
FSA (g/L)	Mean Particle Size (μ m) ^a	Yield (%) ^b
0 (basic)	73, 75	33
0.65	59	87
0.91	54, 55	80
1.0	64, 72, 70, 70	79
2.45	69, 71	82
3.75	62, 57	82
5.64	75, 77, 85	83
7.5	72, 93, 87	79
10.0	37	83
15.0	89, 94, 84	84
20.0	113, 105	71

a Multiple results refer to separate determinations on separate samples taken from the product batch.

b Based on styphnic acid starting material.

TABLE 2

Results for Normal Lead styphnate Type RD1303M
Preparations and Comparison with Production Samples

Batch	Mean Particle Size (μ m) ^a	Yield (%) ^b
1,2/86 (combined)	86,85	not determined
3/86	89,91,111	not determined
9/86	83,91,90	38
10/86	73,73	33
11/86	76,73,71	34
For comparison		
14/78 ^c	111,112	
Batch 34 ex MFF	31	

^a Separate results refer to measurements on separate samples taken from the total batch.

^b Based on styphnic acid.

^c Batch prepared at MRL and used for all the CC Primer developmental work [2,4].

TABLE 3

Results for Preparation of Normal Lead Styphnate RD1302 using Variations on the General Method and Comparison with Production Samples

Batch	Stirrer Speed (rpm)	Acetic Acid (mL/L)	Mean Particle Size (μm) ^a	Yield (%) ^b
27/86	360	-	92,87,87,95	51
28/86	360	-	81,79,77,78	39
31/86	180	-	125,122,121	71
33/86	180	-	90,92	75
34/86	180	-	97,96	62
32/86	100	-	91,83,85,85	70
29/86	360	1.0	87,91,87,89	44
30/86	360	1.8	74 + 137 (bimodal)	44
<u>MFF Samples ^c</u>				
Batch 2099			40	
Batch 2640			33,31	
Batch 2741			68,68	

^a Separate results refer to determinations on separate samples from the total batch.

^b Based on styphnic acid.

^c For comparison of particle size.

TABLE 4

Performance Tests on M52A3B1 Primers Filled with Differing
Normal Lead Styphnates as Conducting Component
and DEFA Priming Mix

	Normal Lead Styphnate ^a			
	RD1302 Batch 34/86	RD1367 Batch 16/86	RD1303M Batch 34 Ex MFF	RD1303M Batch 14/78
Mean particle size (μm)	97.96	75.77, 85	31	111,112
Primer resistance (g)				
When pressed [std. dev.]	4.2[0.6]	4.6[0.5]	8.1[1.35]	
After 2 weeks [std. dev.]	4.4[0.65]	4.95[0.65]	8.4[1.3]	4.3[0.5]
Energy Sensitivity (μJ)				
50% firing probability	4295 ^b 3740 ^c	4415 ^b 4620 ^c	2665 ^b 1950 ^c	2900 ^{c,d}
0.1% 95% conf.	586 ^c	1280 ^c	76.3 ^c	

^a All primers have normal lead styphnate/Tintercarb 140 (96:4) as conducting increment with DEFA priming mix as priming increment.

^b Preliminary Bruceton analysis [15] on 25 primers.

^c Probit analysis [16].

^d These primers were uncapped. Capping leads to increased 50% energies.

TABLE 5

Assessment of Pilot Production Primers EX MFF St Marys and
Normal Lead Styphnate used in their Manufacture

	Production Lot		
	Mix A	Mix B	For Comparison
Normal Lead Styphnate			
Type/Origin	RD1302 MFF Batch 2733 ^a	Batch 4 ex MFF ^b	RD1302, MFF Batch 2753
Particle size distribution (μm)			
Mean	93.3, 96.6, 88.1	92.8, 90.7	92.4, 93.3
Median	79-87	83	85
Upper 16%	> 105	> 100	> 110
Lower 16%	< 62	< 65	< 59
Primer Resistance (g) (std. dev.)	5.5 (0.5)	5.4 (0.5)	
Energy Sensitivity (μJ)			
50% firing energy ^c	8290	8450	
Functioning time (ms) (std. dev.)	0.046 (0.005)	0.047 (0.003)	

a Production batch selected as being of high bulk density and large particle size.

b Modified production batch produced under direction of Mr G. Siddiqui (see text).

c Bruceton Analysis [15] using a 0.2 μF capacitor.

APPENDIX A

AN OUTLINE OF THE MANUFACTURING PROCESS FOR NORMAL
LEAD STYPHNATE RD1302 AS FOLLOWED AT MFF ST MARYS

<u>Raw Material</u>	<u>Quantity</u>	<u>Proportion</u>	<u>Feed Rate</u>
Lead Acetate	14.7 L	300 g/L	2-2.5 min run-in
Magnesium Styphnate pH 4.4	25 L	123.2 g/L (Styphnic acid)	in pan
Nitric Acid	5.1 L	220 g/L	0.34 L/min

Process: Precipitation

Process Parameters: Temperature of Precipitation: 76°C
Stirrer Speed: 70 rpm
Tilt of pan: Nil

Run-in Lead Acetate in 2-2.5 minutes (solution at 75°C)
Stir for 2.5-3 minutes (total time = 5 minutes to this
stage)
Run in Nitric Acid (5.1 L in 15 minutes)
Tilt pan 10° (1.25 turns)
Stir 15 minutes
Cool precipitation vessel to 36°C
Settle two minutes
Wash

Further Finishing Operations:

2 x 40 L water washes in the pan
Precipitate divided into 6 equal portions
Wash under vacuum (water 1 L; methylated spirits
500 mL, vacuum 10" Hg max)
Air drying
Sieving
Weighing

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Normal lead styphnate suitable for Australian production
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ABSTRACT

Normal lead styphnate types RD1302, RD1303M and RD1367 were assessed for their suitability for production and use in Australian manufactured low sensitivity CC primers. Recommendation was made that type RD1302 should be used. The sensitivity penalty incurred by using small particle size lead styphnate has been demonstrated. Pilot lot production of primers at MFF St Marys using selected production RD1302 has verified suitability for the intended application.

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